



Retrieval analysis of lingual fixed retainer adhesives

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Abstract: INTRODUCTION: Our objective was to analyze the surface and bulk properties alterations of clinically aged composites used for fixed retention. **METHODS:** Twenty-six lingual retainers bonded for different time periods (2.2-17.4 years) were retrieved from postorthodontic patients. Fifteen lingual retainers had been cemented by a chemically cured adhesive (Maximum Cure, Reliance Orthodontic Products, Itasca, Ill), and 11 were treated with a photo-cured adhesive (Flow-Tain, Reliance Orthodontic Products). The first group was in service for 2.8 to 17.4 years and the second for 2.2 to 5.4 years. Five specimens from each material were prepared and used as the control (or reference) group. The debonded surfaces from enamel were studied by attenuated total reflectance Fourier transform infrared spectroscopy (n = 3 per material per group), low-vacuum scanning electron microscopy, and energy dispersive x-ray microanalysis (n = 3 per material per group). All specimens were used for the assessment of Vickers hardness, indentation modulus, and elastic index with the instrumented indentation testing method. The values of Vickers hardness, indentation modulus, and elastic index were compared between the retrieved and the reference groups with 1-way analysis of variance and the Student-Newman-Keuls multiple comparison test ($\alpha = 0.05$). **RESULTS:** The attenuated total reflectance Fourier transform infrared spectroscopy analysis showed that both retrieved composites demonstrated reduced unsaturation in comparison with the corresponding reference specimens. Some bonded surfaces showed development of organic integuments. All retrieved specimens showed reduced silicon content. Barium was identified only in the photo-cured group. No significant differences were found between the reference and retrieved groups in Vickers hardness, indentation modulus, and elastic index. **CONCLUSIONS:** Despite the changes in composition, the mechanical properties of the materials tested remained unaffected by intraoral aging.

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Original article

Retrieval analysis of lingual fixed retainer adhesives

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Highlights

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Multistranded wires were bonded to lingual enamel for orthodontic retention.

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Effects of intraoral aging on light- and chemically cured composites were compared.

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Some differences in structure and composition occurred after prolonged exposure.

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Most mechanical properties were unaffected.

Introduction

Our objective was to analyze the surface and bulk properties alterations of clinically aged composites used for fixed retention.

Methods

Twenty-six lingual retainers bonded for different time periods (2.2-17.4 years) were retrieved from postorthodontic patients. Fifteen lingual retainers had been cemented by a chemically cured adhesive (Maximum Cure, Reliance Orthodontic Products, Itasca, Ill), and 11 were treated with a photo-cured adhesive (Flow-Tain, Reliance Orthodontic Products). The first group was in service for 2.8 to 17.4 years and the second for 2.2 to 5.4 years. Five specimens from each material were prepared and used as the control (or reference) group. The debonded surfaces from enamel were studied by attenuated total reflectance Fourier transform infrared spectroscopy (n = 3 per material per group), low-vacuum scanning electron microscopy, and energy dispersive x-ray microanalysis (n = 3 per material per group). All specimens were used for the assessment of Vickers hardness, indentation modulus, and elastic index with the instrumented indentation testing method. The values of Vickers hardness, indentation modulus, and elastic index were compared between the retrieved and the reference groups with 1-way analysis of variance and the Student-Newman-Keuls multiple comparison test ($\alpha = 0.05$).

Results

The attenuated total reflectance Fourier transform infrared spectroscopy analysis showed that both retrieved composites demonstrated reduced unsaturation in comparison with the corresponding reference specimens. Some bonded surfaces showed development of organic integuments. All retrieved specimens showed reduced silicon content. Barium was identified only in the photo-cured group. No significant differences were found between the reference and retrieved groups in Vickers hardness, indentation modulus, and elastic index.

Conclusions

Despite the changes in composition, the mechanical properties of the materials tested remained unaffected by intraoral aging.

Changes in dental arch dimensions due to age are a common occurrence affecting both untreated and orthodontically treated persons. With age, decreases in arch length and width are usually witnessed, causing considerable anterior crowding and increased incisor irregularity.^{1, 2, 3 and 4} Because development of unsatisfactory dental alignment is often the medium- to long-term treatment outcome, there has been a consensus among clinicians that retention should last as long as perfect alignment is desired.^{5, 6 and 7}

Lingual fixed retainers have thus become the only orthodontic device designed to remain bonded over many years and even decades. Based on the prevailing literature, it appears correct to state that no associations between bonded retainers and periodontal diseases could be substantiated.^{8 and 9} Also, no evidence indicates that fixed retention is a cause for increased incidence of dental caries or white spot lesions.¹⁰ Because recent evidence suggests that a bonded retainer may fail to preserve its passivity, the choice of inappropriate wire, material fatigue induced through physiologic masticatory forces, and bonding failure may be sources of developing undesirable effects.^{11 and 12}

It is well known that dental composite resins are subjected to intraoral degradation from exposure to mechanical, thermal, and chemical agents.^{13, 14, 15, 16 and 17} The latter may significantly reduce bond strength,¹⁸ probably through diffusion into the composite-tissue interface.¹⁹

Despite several in-vitro investigations focusing selectively on chemical, thermal, or mechanical stress-related impact, there is limited information on the effects of intraoral aging on composite properties in general, and nothing about intraoral aging of composites used to bond retainers in particular.²⁰

The purpose of this study was to assess the changes in the composition and mechanical properties of composite resins used for bonding orthodontic retainers after intraoral aging. Since differences in the curing mechanism may affect the composite properties, both light-cured and chemically cured materials were analyzed.²¹ The null hypothesis was that there would be no significant differences in the properties of the materials tested between the retrieved and the control samples.

Material and methods

Forty patients finished with orthodontic treatment and retention were selected according to the following inclusion criteria: bonded lingual retainers intended to be removed; no active caries, restorations, or fractures of mandibular anterior teeth; and adequate oral hygiene. The fixed retainers consisted of 0.022 × 0.022-in multistranded stainless steel wires (Tru-Chrome; Rocky Mountain Orthodontics, Denver, Colo) and had been adapted at chairside to fit passively. During bonding, the lingual surfaces of the 6 mandibular anterior teeth had been pumiced, rinsed with water, air-dried, and etched with 37% orthophosphoric acid liquid. The retainers had been bonded to enamel with (1) an unfilled chemically cured, 2-liquid bonding resin and a 2-paste orthodontic adhesive (Maximum Cure and Excel; Reliance Orthodontic Products, Itasca, Ill), and (2) an unfilled light-cured bonding resin and a light-cured orthodontic adhesive (Assure and Flow-Tain; Reliance Orthodontics Products). The compositions of the materials used in the study are listed in Table I.

Table I.

Monomer and filler content of the materials tested as provided by the manufacturer

Ingredient	Excel		Flow-Tain	Maximum Cure: Liquid A	Maximum Cure: Liquid B	Assure
	Paste A	Paste B				
Resin content (wt%)						
BisGMA	-	>20	-	50-80	50-80	-
EBDMA	-	-	>10	-	-	-
TEGDMA	5-15	>5	<15	-	-	-
MMA	-	-		20-40	20-40	-
MMA-F	-	-	-	2-5	-	-
HEMA (stabilized)	-	-	-	-	-	20-30 in acetone
Filler content (wt%)						
Silica	40-70	40-80	-	-	-	-
Glass filler	-	-	>50	-	-	-
Sodium fluoride	<1	-	-	-	-	-

Bis-GMA, Bisphenol glycidyl dimethacrylate; EBPDMA, ethoxylated bisphenol dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; MMA, methyl methacrylate; MMA-F, hydrofluoride methacrylate; HEMA, hydroxyethyl methacrylate.

Approval of the appropriate ethical committee was obtained for the retrieval (University of Athens, number 227, January 20, 2015). Bonding and retrieval of the retainers were performed by the same orthodontist (N.P.); removal of the fixed retainers was not related to the scope of this study. The materials used, the process adopted, and the date of insertion of the fixed retainer were meticulously recorded, and the retrieval process consisted of careful removal of the lingual retainers with a How's pliers. The patient's oral hygiene was assessed before the removal and throughout the maintenance period as well as by examination by the general practitioner.

After retrieval, the materials were forwarded to the laboratory for analysis. The enamel bonded surfaces of all composite retainer elements were examined under a stereomicroscope (M80; Leica, Wetzlar, Germany) at 16-times magnification. From the retrieved retainers, 15 lingual retainers bonded with Excel (EX-INV) and 11 with Flow-Tain (FT-INV) (total, 26) were collected; the remaining were excluded for reasons related to the lack of adequate mass of adhesive attached to the wires, broken adhesive, cohesive failure of the adhesive on the enamel, and fully or partially debonded adhesive from the enamel attached to the retainer. The intraoral aging periods ranged from 2.8 to 17.4 years for the EX-INV retainers and from 2.2 to 5.4 years for the FT-INV retainers.

Specimens of each adhesive material were cured in vitro as follows. Microscopic glass slides were covered with transparent polyester film strips and received 2 spacers (1.5 mm), 1 on each side. The adhesive resin pastes (approximately 2 mm in length \times 2 mm in width; $n = 3$) were placed and covered with a second strip and slide. For the light-cured material, photopolymerization was performed with a light-emitting diode curing unit (Bluephase G2; Ivoclar Vivadent, Schaan, Liechtenstein) emitting 1.2 W per square centimeter of irradiance at the standard irradiation mode for 20 seconds. These specimens were stored in an incubator at 37°C for 7 days under dark and dry conditions, and were used as references for Excel (EX-REF) and Flow-Tain (FT-REF), respectively.

Attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) was performed on the composite surfaces debonded from the lingual enamel surfaces to identify the molecular compositions and the degrees of carbon double-bond conversion. Three specimens from each retrieved set and control group were analyzed, to characterize the changes in molecular composition. Spectra acquisitions were obtained with a Fourier transform infrared spectrometer (Spectrum GX; PerkinElmer, Baconsfield, United Kingdom), using a microattenuated total reflection accessory (Golden Gate; Specac, Fort Washington, Pa) with a 1 \times 1 mm diamond reflective element under the following conditions: 4000 to 650 cm^{-1} range, 4 cm^{-1} resolution, 20 scans coaddition, and 2- μm depth of analysis at 1000 cm^{-1} . All spectra were subjected to attenuated total reflection and baseline corrections. The peak absorbance height ratio of the aliphatic C = C (1638 cm^{-1}) to aromatic C..C (1608 cm^{-1}) groups used to measure the degree of cure was used to calculate the percentage differences in the degree of cure between the retrieved and the control specimens.²²

The same samples were further investigated for their structure and elemental composition, using a variable pressure scanning electron microscope (Quanta 200; FEI, Hillsboro, Ore) with a large field detector for secondary electron imaging and a solid-state detector for backscattered electron imaging. The operating parameters were 1 Pa chamber pressure (low-vacuum, 20 kV beam acceleration voltage, 105 μA beam current, and 800-times nominal magnification). The elemental composition was investigated by x-ray energy dispersive microanalysis. Spectra were collected using an X Flash 6|10 silicon drift detector (Bruker, Berlin, Germany) with a slew window under the same operating conditions, using a 64 \times 64- μm sampling window and 200-second acquisition time. The elemental quantification was carried out in a standardless mode using atomic number, absorbance, and fluorescence correction factors.

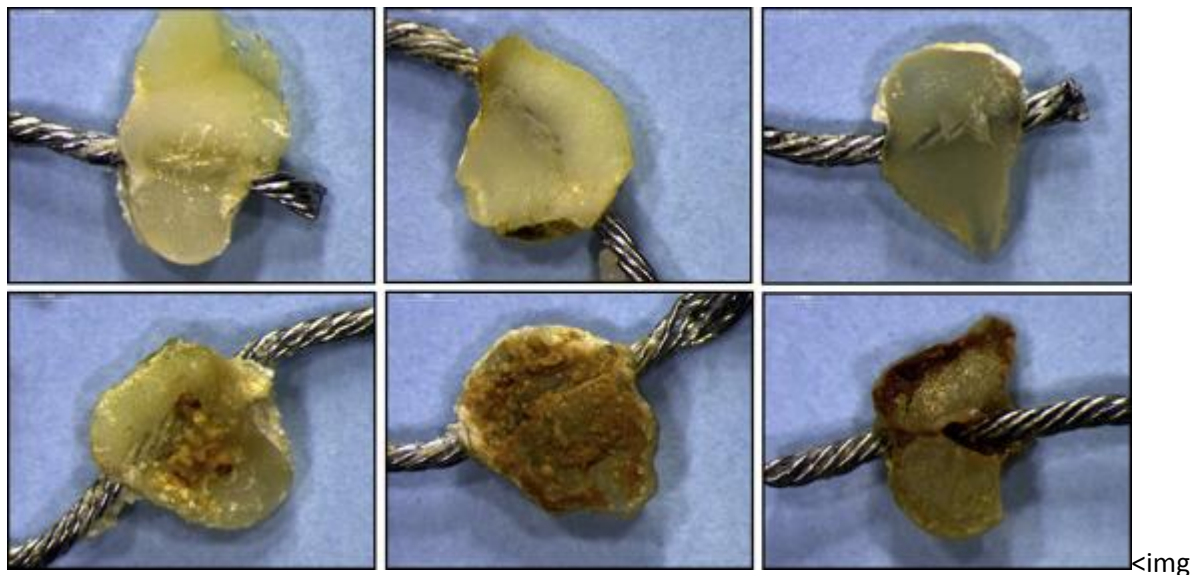
All specimens were embedded in self-cure acrylic resin (10 mm thick) and, after metallographic grinding and polishing (300-1200 grit-size silicon-carbon papers, under water rinsing plus 5 minutes of sonication in water), were subjected to instrumented indentation testing with a Vickers indenter. One force-indentation depth curve was recorded from each specimen under a 4.9-N load and a 2-second dwell time, using a universal hardness testing machine (ZHU 0.2/Z2.5; Zwick Roell, Ulm, Germany). The indentation modulus, elastic index (defined as the elastic [area under the curve at the elastic limit] to total work ratio), and Vickers hardness (HV5) were determined according to the ISO 14577-1 specification.²³

Statistical analysis

The values of the indentation modulus, elastic index, and Vickers hardness were statistically analyzed by 1-way analysis of variance with the independent parameter the aging status (in-vivo aged, control). The Student-Newman-Keuls test was used for multiple comparisons. For the percentage differences in the degree of cure, an unpaired t test was used. Data normality and equal variance were assessed by the Kolmogorov-Smirnov and Levene tests, respectively. Statistical analysis was performed by SigmaStat software (version 3.1; Systat Software, San Jose, Calif).

Results

Figure 1 illustrates the stereoscopic images of retrieved composite surfaces bonded to enamel. Clear debonded surfaces were used for the analysis, whereas contaminated surfaces were excluded from the study.

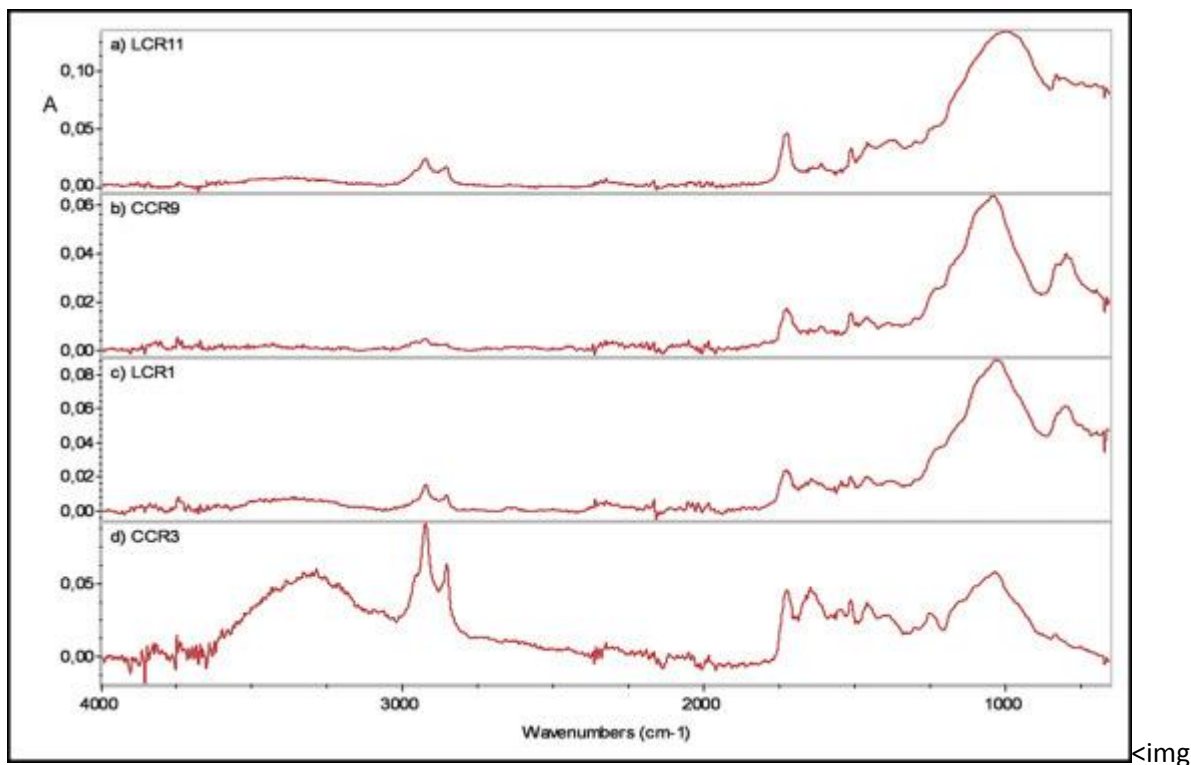


class="figure large" border="0" alt="Stereomicroscopic images of debonded composite specimens with clean (top row) ..." src="http://origin-ars.els-cdn.com/content/image/1-s2.0-S0889540616302906-gr1.jpg" data-thumbEID="1-s2.0-S0889540616302906-gr1.sml" data-imgEIDs="1-s2.0-S0889540616302906-gr1.jpg" data-fullEID="1-s2.0-S0889540616302906-gr1.jpg">

Fig 1.

Stereomicroscopic images of debonded composite specimens with clean (top row) and contaminated (bottom row) surfaces. Contaminated surfaces at such extents were excluded from the study (16-times magnification; bar = 1 mm).

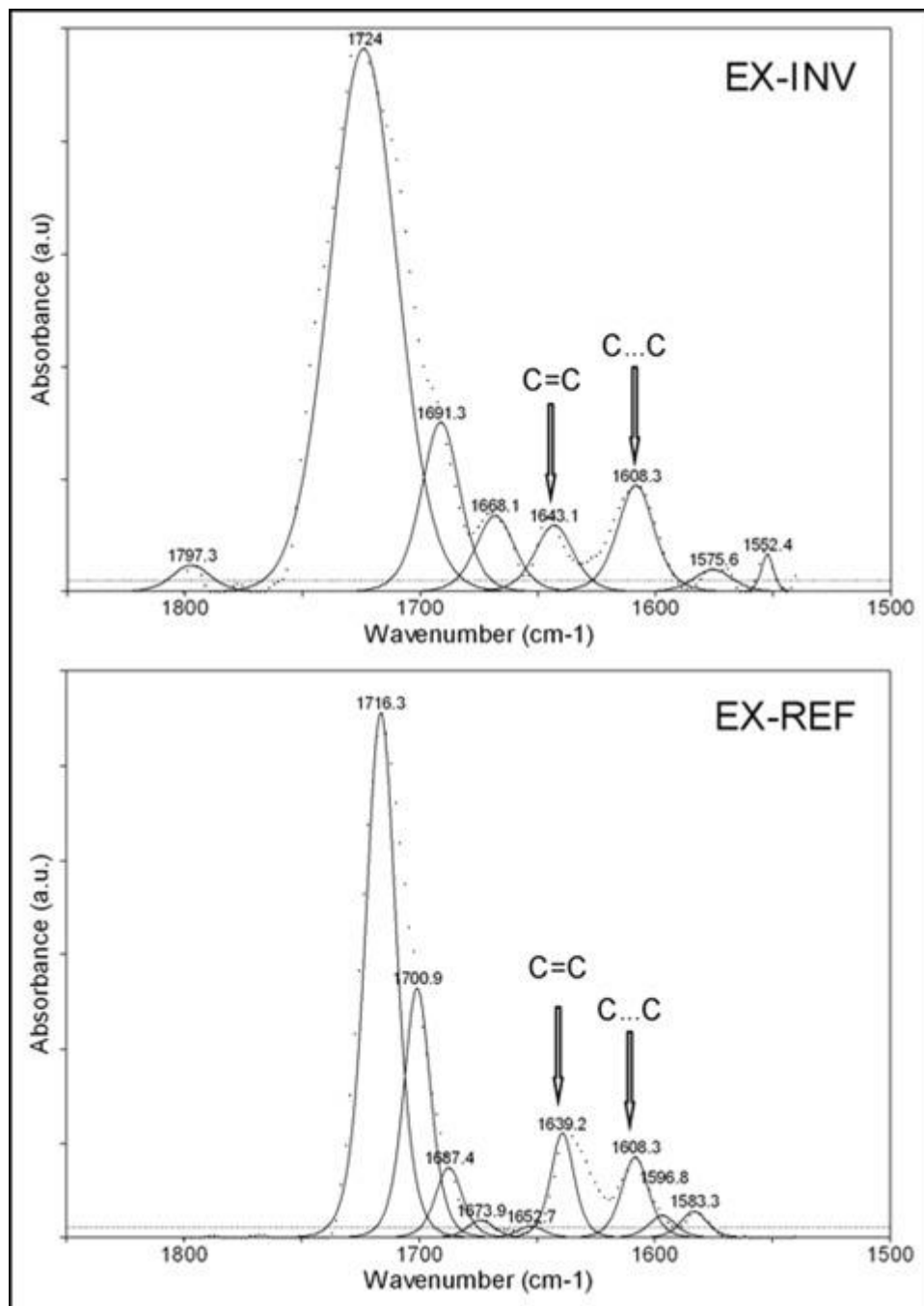
Representative ATR-FTIR spectra from the reference and in-vivo aged materials are shown in Figure 2. The peak assignments include the following groups: OH free (3746 cm^{-1}), OH hydrogen bonded (3380 cm^{-1}), CH_2/CH_3 ($2920, 2853, 1465\text{--}1430\text{ cm}^{-1}$), $\text{C}=\text{O}$ ($1724, 1320, 1290\text{ cm}^{-1}$), $\text{C}=\text{C}$ ($1636, 980\text{--}950, 809\text{ cm}^{-1}$), aromatic $\text{C}\cdot\text{C}$ ($3060, 1608, 1510, 770, 700\text{ cm}^{-1}$), aromatic- $\text{C}-\text{CH}$ (1300 cm^{-1}), aromatic- $\text{O}-\text{CH}$ (1250 cm^{-1}), $\text{CH}-\text{OH}$ ($1180\text{--}1160\text{ cm}^{-1}$), $\text{C}-\text{O}-\text{C}$ ($1100\text{--}1050\text{ cm}^{-1}$), and $\text{Si}-\text{O}-$ ($1080\text{--}1020, 790\text{--}800\text{ cm}^{-1}$), the latter attributed to silica and glass-filler interferences (Fig 2, A-C). One specimen demonstrated organic (proteinaceous) contamination (Fig 2, D) as evidenced by the increased relative intensity of the peaks at $3500\text{ to }3000\text{ cm}^{-1}$ (OH and N-H), 1740 cm^{-1} ($\text{C}=\text{O}$), 1650 cm^{-1} ($\text{C}=\text{O}$ of amide I), 1540 and 1250 cm^{-1} ($\text{C}=\text{O}$, N-H of amide II and III, respectively), and $1200\text{ to }1100\text{ cm}^{-1}$ ($\text{C}-\text{OH}$). The spectra of the retrieved materials showed a greater contribution of the $\text{Si}-\text{O}-$ peak relative to the $\text{C}=\text{O}$ component. Expanded spectra of a retrieved (Fig 3, EX-INV) and a control (Fig 3, EX-REF) adhesive at the region used for assessment of the degree of cure are shown in Figure 3. The results of the percentage differences in the degree of cure were (mean, standard deviation): Excel = $+10.2 (3.1)\%$ and Flow-Tain = $+7.6 (2.4)\%$, with the positive values indicating higher conversion in the in-vivo aged materials. No statistically significant difference was found between the 2 means ($P > 0.05$).



class="figure large" border="0" alt="Representative ATR-FTIR spectra from the in-vivo aged groups. R, Retrieved; LC, ..." src="http://origin-ars.els-cdn.com/content/image/1-s2.0-S0889540616302906-gr2.jpg" data-thumbEID="1-s2.0-S0889540616302906-gr2.sml" data-imgEIDs="1-s2.0-S0889540616302906-gr2.jpg" data-fullEID="1-s2.0-S0889540616302906-gr2.jpg">

Fig 2.

Representative ATR-FTIR spectra from the in-vivo aged groups. R, Retrieved; LC, light cured; CC, chemically cured. Note the organic contamination in spectrum d (all in absorbance scale).



Expanded ATR-FTIR spectra from in-vivo aged (top) and reference (bottom) ...
src="http://origin-ars.els-cdn.com/content/image/1-s2.0-S0889540616302906-gr3.jpg" data-thumbEID="1-s2.0-S0889540616302906-gr3.sml" data-imgEIDs="1-s2.0-S0889540616302906-gr3.jpg" data-fullEID="1-s2.0-S0889540616302906-gr3.jpg">

Fig 3.

Expanded ATR-FTIR spectra from in-vivo aged (top) and reference (bottom) specimens of Excel. Arrows show the aliphatic C = C and aromatic C..C peaks used for calculation of the percentage differences in the degree of conversion measurements (absorbance scale, 1850-1500 cm^{-1} wave number range).

Secondary electron and backscattered electron images from the reference and retrieved Excel material are depicted in Figure 4. The backscattered electron images clearly demonstrate the filler particles with irregular shapes and higher mean atomic numbers (lighter gray scale). The main differences between the 2 images were the increased porosity in the retrieved material and the presence of some fractured ridges associated with the debonding procedure. The corresponding images for the Flow-Tain material (FT-REF and FT-INV) are illustrated in Figure 5. The smooth morphology of the control surface demonstrated few regions with lighter gray scale, assigned to filler particles. The retrieved surfaces, though, demonstrated a complex pattern with microporosity, pitting, and cracking.



Fig 4.

Secondary electron and backscattered electron images from the surfaces of EX-REF): A, secondary electron image; B, backscattered electron image; and EX-INV; C, secondary electron image; D, backscattered electron image (original magnification, 800 times; bar = 100 μm).

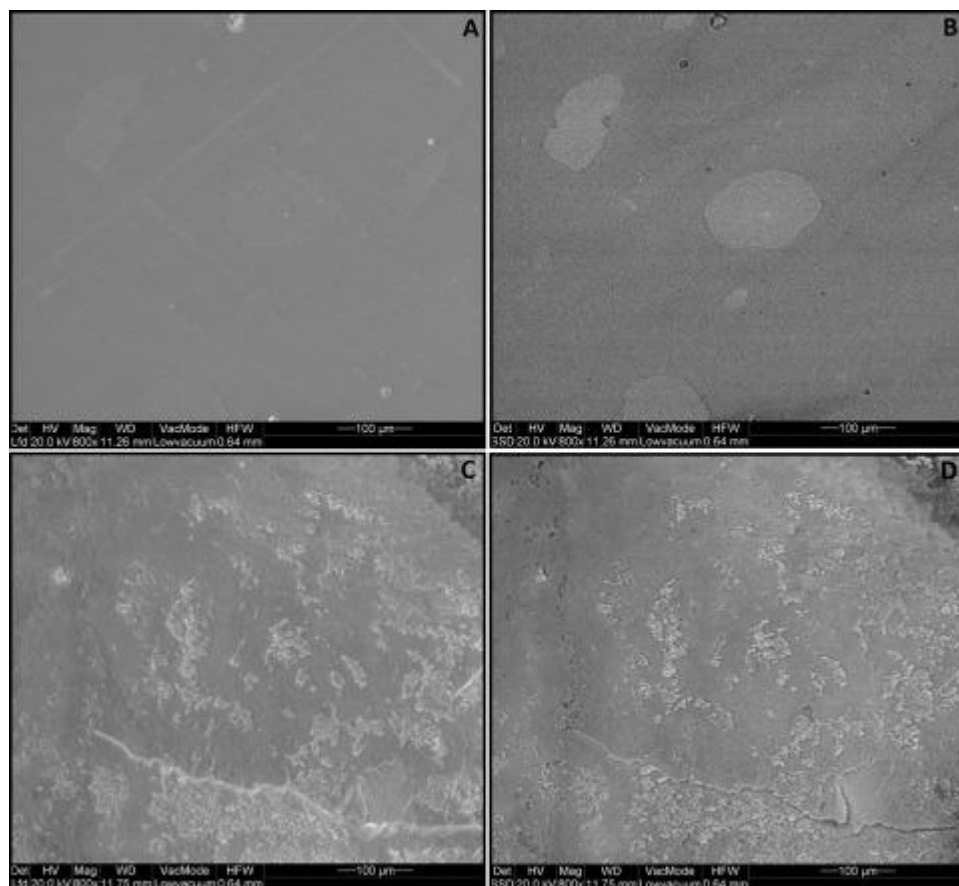


Fig 5. Secondary electron and backscattered electron images from the surfaces of EX-REF: A, secondary electron image; B, backscattered electron image; and EX-INV: C, secondary electron image; D, backscattered electron image (original magnification 800 times; bar = 100 μm).

Secondary electron and backscattered electron images from the surfaces of FT-REF: A, secondary electron image; B, backscattered electron image; and FT-INV: C, secondary electron image; D, backscattered electron image (original magnification 800 times; bar = 100 μm).

Control and retrieved energy dispersive microanalysis spectra per material are presented in Figure 6. The corresponding qualitative and quantitative results are summarized in Table II. The elements phosphorus, chlorine, silicon, potassium, and calcium, which appeared only in the retrieved materials, are exclusively attributed to the oral environment. The weight percent (wt%) differences in the major material components (carbon, oxygen, silicon, and barium) may indicate regional variations in composition caused by changes in resin matrix or filler particle content (ie, absorption of carbohydrates,

presence of unfilled bonding resin, and so on). For elements with minor content (sodium, aluminum, titanium), the wt% increase in the retrieved materials should be mostly attributed to contaminants from the oral environment (ie, toothpastes with aluminum and titanium).

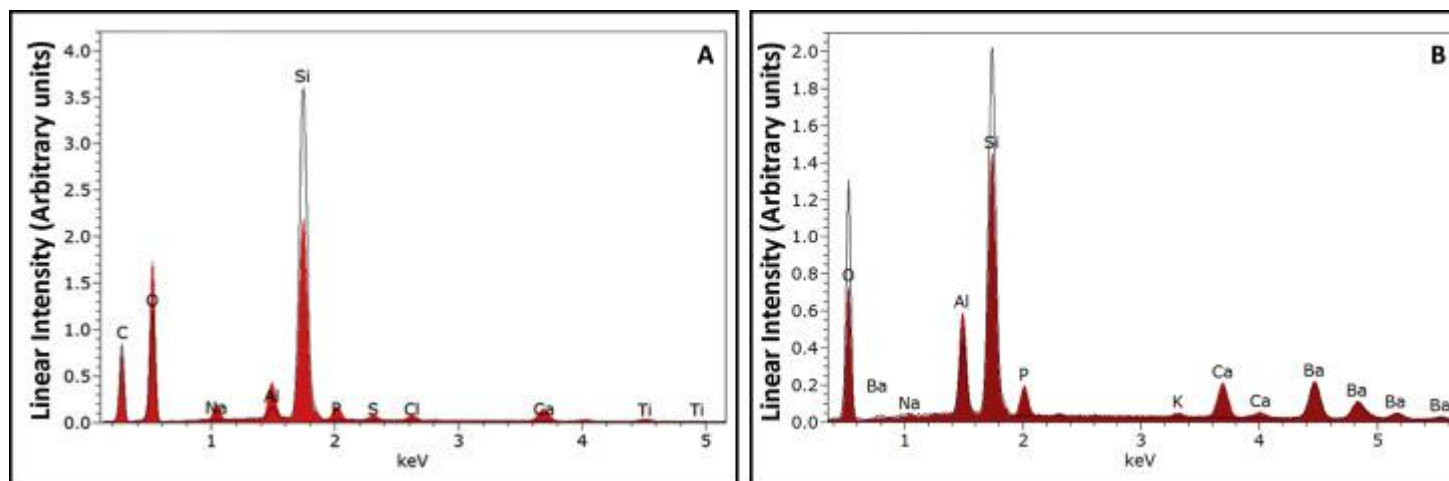


Fig 6.

Energy dispersive analysis spectra from the surfaces of the control and retrieved groups: A, EX-REF (black line) and EX-INV (red area); B, FT-REF (black line) and FT-INV (red area).

Table II.

Elemental composition of the groups tested after energy dispersive analysis (mean values, n = 3, wt%)

Groups	C	O	Na	Al	Si	P	Cl	S	K	Ca	Ti	Ba
EX-REF	28.8	37.4	0.1	1.0	22.8							
EX-INV	30.2	47.2	1.9	2.8	13.1	1.2	0.5	0.4		2.0	0.6	
FT-REF	38.0	35.5	0.1	2.9	13.2							10.4
FT-INV	42.1	26.4	0.2	4.5	10.2	1.3			0.1	3.2		12.3

C, Carbon; O, oxygen; Na, sodium; Al, aluminum; Si, silicon; P, phosphorus; Cl, chlorine; S, sulfur; K, potassium; Ca, calcium; Ti, titanium; Ba, barium.

In Figure 7, the force-indentation depth curves for each material are presented. The results of mechanical properties derived from instrumented indentation testing are summarized in Table III. No statistically significant differences were found in the aging status (control or in-vivo aged) for the materials in all properties tested. However, statistically significant differences were found between the 2 materials. Excel showed higher indentation modulus and Vickers hardness values but similar a elastic index compared with Flow-Tain.

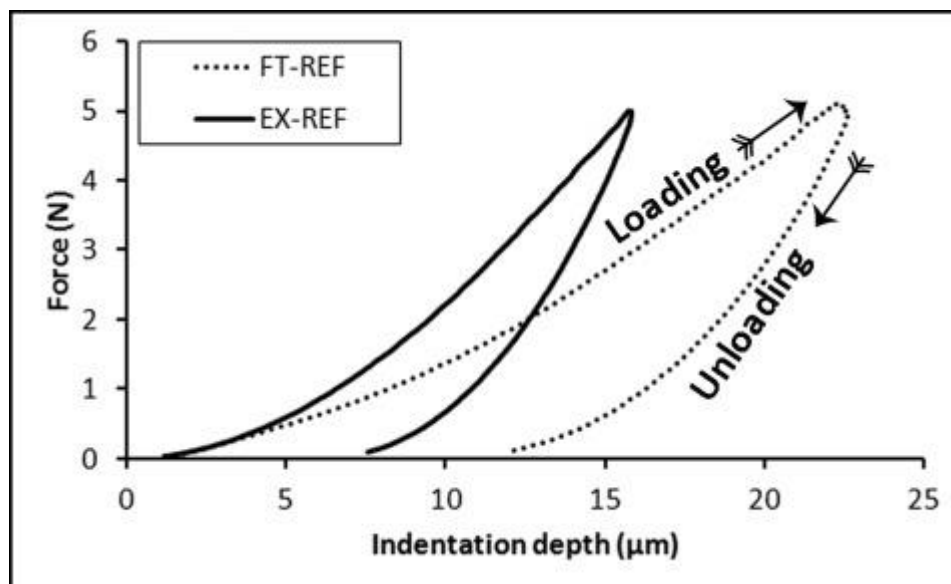


Figure 7. Force-indentation depth curves from the reference materials included in the study.

Fig 7.

Force-indentation depth curves from the reference materials included in the study.

Table III.

Means and standard deviations (in parentheses) of mechanical properties tested

Group	EIT (GPa)	η_{IT} (%)	HV
EX-REF	8.3 (0.7) ^a	44 (2.4) ^a	70 (5) ^a
EX-INV	8.8 (1.4) ^a	47 (3.7) ^a	79 (6.4) ^a
FT-REF	5.2 (0.3) ^b	45 (1.4) ^a	37 (1.4) ^b
FT-INV	4.7 (0.3) ^b	46 (1.3) ^a	36 (3.4) ^b

Same superscripts indicate mean values without statistically significant differences ($P > 0.05$).

EIT, Indentation modulus; η IT, elastic index; HV, Vickers hardness.

In this investigation, we aimed at clarifying the impact of intraoral aging on light-cured and chemically cured composites used for orthodontic retention, as bonding elements of multistranded wires to lingual enamel. For the period tested, some differences were encountered in structure and composition after prolonged intraoral exposure, but the bulk mechanical properties remained unaffected. Therefore, the null hypothesis must be partially accepted.

Intraoral aging may affect the structural integrity and influence an important array of material properties, including mechanical performance,²⁴ hydrolytic stability,²⁵ and ²⁶ corrosion, degradation resistance to the intraoral biochemical environment,²⁴ and ²⁷ and aggressive chemical stimuli (ie, fluorides, bleaching agents, alcohol, and so on). Despite numerous efforts to simulate intraoral aging procedures in laboratory testing, the results of retrieval analysis are considered more reliable to show a positive or negative treatment effect.

The composite surfaces analyzed were those debonded from enamel and not directly exposed to the oral environment, since the integuments formed on the latter may hinder the analysis of subsurface material. This is critical when the acquired film thickness exceeds the mean sampling depth of the method used. Moreover, debonding of the retainers from enamel, a failure mode with frequent incidence,²⁸ is mostly associated with the quality of the enamel-composite interface, which merits further investigation. The quality of acid etching may affect the interfacial bonding, especially when conventional bonding resins are used without monomers functionalized with acidic groups (ie, phosphates, carboxylates). However, this may be implicated with early failures. It is reasonable to expect that the failures after prolonged intraoral service are associated with aging of the weak unfilled bonding resins and the main adhesive composites caused by the applied stresses (mechanical, thermal) and the chemical environment. However, since the composite adhesives used for retainer bonding were directly exposed to the oral environment, such aging effects are expected to the bonded materials as well. A chemically cured material and a light-cured material were used in the study to explore the role of initiation mechanism as well, since poor cross-linking of the polymer network is associated with the release of residual monomers.²⁹

The ATR-FTIR analysis in the retrieved materials demonstrated increased noise because the debonded surfaces were not perfectly smooth, which prevented good contact with the reflective diamond element. The debonded surfaces, apart from one, were free of intraoral integuments that could interfere with the chemical groups of the materials, especially with those used for assessment of the percentage differences in the degree of cure. The shielding effect of the proteinaceous material adsorbed onto the adhesive is clearly demonstrated in Figure 2, D, where the original adhesive peaks are completely masked off by a material of proteinaceous origin. The increased degree of cure of the retrieved materials may be explained by an increased cross-linking or C = C degradation due to aging. ³⁰ Since the adhesive pastes were bonded to enamel with the corresponding unfilled resins, a question is raised on the validity of using the pastes as references for calculation of the percentage differences in the degree of cure. Spectra of set and unset adhesive pastes and unfilled bonding resins were used to allocate spectral differences to identify the presence of a principal component in the debonded

surfaces. The strong and broad peak of Si-O- ($1080\text{--}1020\text{ cm}^{-1}$) was the most characteristic of the silicon dioxide filler content in the pastes and used to probe the exposure of composite pastes in the debonded specimens. This peak was found in all debonded specimens analyzed by ATR-FTIR.

Excel adhesive is more heavily filled than is Flow-Tain, and this agrees with the Vickers hardness and elastic modulus findings, as measured with instrumented indentation testing. The presence of silicon and the traces of sodium and aluminum are appended to the filler component. Carbon is mostly associated with the organic matrix and oxygen with the organic matrix and the oxides of the exposed filler particles. The surfaces of the retrieved materials had protruding filler particles from the organic matrix because of disruption of the organic phase during debonding and polishing. The material also showed a random distribution of pores, caused by air entrapment during hand mixing of the 2 paste components.

The presence of silicon, barium, aluminum, and sodium in Flow-Tain is attributed to the filler system, carbon to the organic matrix, and oxygen to both the organic matrix and the inorganic oxide compounds. A random distribution of pits was identified on the surfaces of the retrieved specimens. The traces of phosphorus, potassium, and calcium should be rather implicated with enamel microfragments produced during debonding. The differences found between the retrieved and control specimens in the elemental composition of the native materials were limited to a strong silicon reduction in Excel and a smaller one in Flow-Tain, with a concurrent increase in the other elements.

A further aim of this study was to investigate whether intraorally aged composites used for fixed retention show differences in their mechanical properties. A recent article demonstrated significant dissimilarities in the conversion rates of intraorally aged resin composites compared with specimens stored in water³⁰; it was hypothesized that the mechanical properties of retrieved specimens may be significantly altered after aging in the oral environment. Testing of the mechanical properties of retrieved samples with conventional tests (compression, tension, bending, and so on) has been constrained by the need of bulky specimens, which are not obtainable for dental materials. This specific limitation has been overcome by a method recently introduced (instrumented indentation testing), which allows the assessment of a variety of mechanical properties such as hardness, modulus, elastic index, creep, and so on, to be analyzed in small samples.²³

Changes in the surface composition and conversion did not substantially influence the mechanical properties of the retrieved adhesives, which were registered at near-surface regions, after controlled grinding and polishing. Noteworthy was nonetheless the observed difference between samples of the 2 materials, since Excel produced significantly higher indentation modulus and indentation hardness values compared with Flow-Tain. This difference should be assigned to the higher inorganic filler content and the light-curing polymerization mechanism of the former. Increased indentation modulus is desirable because equal resistance to stress can be provided with smaller cross sections. Higher hardness values, indicating greater wear, are equally advantageous. Both materials demonstrated similar elastic index values, which indicated a similar elastic work in the reference and aged states.

Conclusions

Intraoral aging may influence the chemistry of the resin composite adhesives used for lingual fixed retainer bonding but does not substantially affect their modulus of elasticity, hardness, and elastic work.

References

1

J.M. Felton, P.M. Sinclair, D.L. Jones, R.G. Alexander

A computerized analysis of the shape and stability of mandibular arch form

Am J Orthod Dentofacial Orthop, 92 (1987), pp. 478–483

2

R.A. Riedel, S. Brandt

Dr. Richard A. Riedel on retention and relapse

J Clin Orthod, 10 (1976), pp. 454–472

3

B.U. Zachrisson

Important aspects of long-term stability

J Clin Orthod, 31 (1997), pp. 562–583

4

A. de la Cruz, P. Sampson, R.M. Little, J. Årtun, P.A. Shapiro

Long-term changes in arch form after orthodontic treatment and retention

Am J Orthod Dentofacial Orthop, 107 (1995), pp. 518–530

5

F.A. Booth, J.M. Edelman, W.R. Proffit

Twenty-year follow-up of patients with permanently bonded mandibular canine-to-canine retainers

Am J Orthod Dentofacial Orthop, 133 (2008), pp. 70–76

6

L.F. Andrews

The six keys to normal occlusion

Am J Orthod, 62 (1972), pp. 296–309

7

W.S. Parker

Retention—retainers may be forever

Am J Orthod Dentofacial Orthop, 95 (1989), pp. 505–513

8

P. Dietrich, R. Patcas, N. Pandis, T. Eliades

Long-term follow-up of maxillary fixed retention: survival rate and periodontal health

Eur J Orthod, 37 (2015), pp. 37–42

9

E.E. Heier, A.A. De Smit, I.A. Wijgaerts, P.A. Adriaens

Periodontal implications of bonded versus removable retainers

Am J Orthod Dentofacial Orthop, 112 (1997), pp. 607–616

10

E.H. Dahl, B.U. Zachrisson

Long-term experience with direct-bonded lingual retainers

11

C. Katsaros, C. Livas, A.M. Renkema

Unexpected complications of bonded mandibular lingual retainers

Am J Orthod Dentofacial Orthop, 132 (2007), pp. 838–841

12

I. Sifakakis, T. Eliades, C. Bourauel

Residual stress analysis of fixed retainer wires after in vitro loading: can mastication-induced stresses produce an unfavorable effect?

Biomed Tech (Berl), 60 (2015), pp. 617–622

13

R.S. Hobson, J.F. McCabe, S.D. Hogg

The effect of food simulants on enamel-composite bond strength

J Orthod, 27 (2000), pp. 55–59

14

E.A. Munchow, A.C. Ferreira, R.M. Machado, T.S. Ramos, S.A. Rodrigues-Junior, C.H. Zanchi

Effect of acidic solutions on the surface degradation of a micro-hybrid composite resin

Braz Dent J, 25 (2014), pp. 321–326

15

C.G. Matasa

Microbial attack of orthodontic adhesives

Am J Orthod Dentofacial Orthop, 108 (1995), pp. 132–141

16

Y. Momoi, H. Iwase, Y. Nakano, A. Kohno, A. Asanuma, K. Yanagisawa

Gradual increases in marginal leakage of resin composite restorations with thermal stress

J Dent Res, 69 (1990), pp. 1659–1663

17

G.G. Montes, R.A. Draughn

In vitro surface degradation of composites by water and thermal cycling

Dent Mater, 2 (1986), pp. 193–197

18

S.Y. Lee, H.M. Huang, C.Y. Lin, Y.H. Shih

Leached components from dental composites in oral simulating fluids and the resultant composite strengths

J Oral Rehabil, 25 (1998), pp. 575–588

19

S.Y. Lee, E.H. Greener, H.J. Mueller

Effect of food and oral simulating fluids on structure of adhesive composite systems

J Dent, 23 (1995), pp. 27–35

20

T. Eliades, C. Bourauel

Intraoral aging of orthodontic materials: the picture we miss and its clinical relevance

Am J Orthod Dentofacial Orthop, 127 (2005), pp. 403–412

21

J.L. Ferracane, E.H. Greener

The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins

J Biomed Mater Res, 20 (1986), pp. 121–131

22

T. Eliades, G. Eliades, W.A. Brantley, W.M. Johnston

Polymerization efficiency of chemically cured and visible light-cured orthodontic adhesives: degree of cure

Am J Orthod Dentofacial Orthop, 108 (1995), pp. 294–301

23

ISO 14577-1:2002. Metallic materials—instrumented indentation test for hardness and materials parameters, International Organization for Standardization, Geneva, Switzerland (2002)

24

W. Wu, J.E. McKinney

Influence of chemicals on wear of dental composites

J Dent Res, 61 (1982), pp. 1180–1183

25

I.E. Ruyter, S.A. Svendsen

Remaining methacrylate groups in composite restorative materials

Acta Odontol Scand, 36 (1978), pp. 75–82

26

L. Papagiannoulis, J. Tzoutzas, G. Eliades

Effect of topical fluoride agents on the morphologic characteristics and composition of resin composite restorative materials

J Prosthet Dent, 77 (1997), pp. 405–413

27

E.C. Munksgaard, M. Freund

Enzymatic hydrolysis of (di)methacrylates and their polymers

Scand J Dent Res, 98 (1990), pp. 261–267

28

A. Iliadi, D. Kloukos, N. Gkantidis, C. Katsaros, N. Pandis

Failure of fixed orthodontic retainers: a systematic review

J Dent, 43 (2015), pp. 876–896

29

K. Inoue, I. Hayashi

Residual monomer (Bis-GMA) of composite resins

J Oral Rehabil, 9 (1982), pp. 493–497

A. Iliadi, S. Baumgartner, A.E. Athanasiou, T. Eliades, G. Eliades

Effect of intraoral aging on the setting status of resin composite and glass ionomer orthodontic adhesives

Am J Orthod Dentofacial Orthop, 145 (2014), pp. 425–433